A NOTE ON THE REFRACTIVE INDEX OF SHELLAC*

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INTRODUCTION

ABSTRACT. Refractive indices of various samples of shellac, lac constituents, and a few other natural and synthetic resins have been determined using the ordinary type of Abbe Refractometer by a simple technique at different temperatures between 20°C and 90°C. From the curves representing the variation of refractive indices of these with temperature, their softening ranges have been found and implications discussed. The temperature coefficients have also been calculated for the different linear portions. It has been suggested in the paper that the determination of refractive indices of all low melting resins may be rapidly and easily made by adopting this simple technique instead of the hitherto followed lens and reflection-type refractomer methods. Houwink's observation that the refractive index of shellac increases on polymerisation by heat has also been confirmed.

The refractive index is one of the most important optical properties which are frequently used for the identification of various solids, liquids and gaseous substances or for the testing of their purity. The appearance of varnish films and paint coatings depends to a large extent on this important characteristic. The relative refractive indices of some fillers used in lacquer and rubber industries are responsible for their transparency. In fact, even most of the non-crystalline substances as for example, artificial silk fibres, wool, cotton, pigments, etc., are also subjected to an examination in regard to their refractive indices.

Bradley [1] has shown the importance of the application of refractive index in the case of resins. He used oblique illumination immersion method and found out correlation of this characteristic of resins with their general physical and chemical properties as a basis for their identification. By using a crystal refractometer Greger [2] also correlated refractive indices of resins with their general physical properties, as for example, density, hardness, melting point, solubility, etc. But the absence of an accurate and easy method, such as the use of a compact and full-proof instrument like the Abbe Refractometer, has been regretted by many workers.[3] Hanstock,[4] therefore, devised a simple method of determining refractive indices of resins by measuring the focal length of a combination of

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two convex lenses and interposing later on the resinous substance in the form of a concave lens cast between the above two convex lenses. Houwink[5] made use of the immersion method and determined the refractive indices of many synthetic and a few natural resins before and after their polymerisation by heating. His study has thrown some light on the structure of these substances. The Hanstock lens method has been used by Thakur and Aldis[6] as well as Verman[7] for a comprehensive study of various lac samples and their constituents. But, though the lens method is simple, however, in principle, some have admitted that it is 'cumbersome' for occasional examination. Very recently West[8] has published several methods of increasing the distinctness of the border line in the field of view of the Abbe Refractometer when the reflection method is used for the measurement of refractive index of resins and plastic bodies. To increase the contrast between the two halves of the field of view he has used polarised light, a polarizing screen between the source of illumination and the front prism as as well as a cap analyser over the eye piece. Though he has claimed some practical advantage for this polarising method, there cannot be any doubt about the superiority of the method of measurement by the transmitted light in the Abbe Refractometer. The object of the present paper is to show that the standard type of the Abbe refractometer can be used for the determination of the refractive index of all low melting resins, natural or synthetic, by a simple technique to eliminate the obvious difficulties, and also to provide correct data for various lac samples corresponding to sodium light at different temperatures. The range through which the refractive index of a genuine lac sample varies has been investigated and discussed at length by Thakur and Aldis[6] and so it was not undertaken by the present author.

EXPERIMENTAL

A Zeiss refractometer with heatable prisms was used for the determination of refractive index of various samples of lac and a few other natural and synthetic resins. An arrangement was made for continuously passing water at any desired temperature through the refractometer prisms with the usual spiral heater and adjustable water-pressure regulator, which are generally supplied with the instrument. Any other arrangement, such as, for example, the circulation of a liquid from a thermostat through the prisms, might have been used. The thermometer usually supplied with the instrument has a graduation up to 75°C. This was replaced by an ordinary thermometer reading up to 100°C.

A small quantity of the resin was then slowly melted on a crucible and carefully applied on the surface of the lower prism, which was kept at or slightly above the melting point of the resin. It has been found that at this temperature the substance spreads itself quite satisfactorily and small air bubbles that may sometimes get entangled in the molten resin during application on the prism surface are easily eliminated if some time (1 or 2 mins.) is allowed to elapse before the prisms are closed. Readings may be taken at any lower temperature now, provided the cooling is gradual and slow. A sudden cooling may produce fine cracks in the resin film resulting in the indistinctness of the line of separation. Moreover, this may put excessive strain on the expensive prisms, though gradual heating or cooling will not affect them. A strong source of illumination facilitates easy and rapid working. A 100-watt, 220-volt opal glass lamp and, also, Bausch and Lomb microscope lamp were used successively by the author with success. A piece of black paper as a screen in front of the compensator to shut off extraneous light was found to give better contrast. Refractive indices were thus determined at different temperatures between 20° C and 100° C. In order to open the prisms for cleaning, the temperature was always raised above the melting point of the resin, when these could be opened and cleaned easily with some cotton wool soaked in a suitable solvent.

RESULTS

The results of refractive-index measurement by this method have been shown in Tables I and II. Data on various samples of lac at different temperatures have been incorporated in Table I, whilst those of lac constituents and other resins in Table II. For the sake of comparison with other available data a separate table (Table III) has been compiled with references. Table IV shows the temperature coefficients of refractive indices.

TABLE I

Lac samples	Refractive Index at							
	20° C	30° C	40° C	50° C	60° C	70° C	80° C	90° C
Kusum shellac	1·5224	1·5210	1·5192	1·5170	1·5130	1·5050	1·5014	1·4980
Palas shellac	1·5235	1·5220	1·5200	1·5172	1·5128	1·5075	1·5035	1·5000
Khair shellac	1·5210	1·5195	1·5175	1·5158	1·5116	1·5070	1·5022	1·4982
Ber shellac	1·5236	1·5225	1·5215	1·5195	1·5152	1·5110	1·5070	1·5026
Pakur shellac	1·5236	1·5222	1·5206	1·5175	1·5136	1·5096	1·5054	1·5012
Button lac (R.L.)	1·5242	1·5222	1·5200	1·5165	1·5126	1·5086	1·5042	1·5005
Dewaxed shellac Blonde	2·5228	1·5200	1·5170	1·5132	1·5090	1·5048	1·5015	1·4976
Shellac Std. I.	1·5250	1·5232	1·5212	1·5180	1·5124	1·5080	1·5040	1·5006
Shellac (superfine unarsenicated)	1·5244	1·5222	1·5200	1·5164	1·5110	1·5070	1·5030	1·4992
Shellac (Superfine arsenicated)	1·5242	1·5225	1·5205	1·5172	1·5128	1·5078	1·5036	1·5000
Shellac (Fine grade)	1·5250	1·5232	1·5212	1·5180	1·5136	1·5195	1·5056	1·5018
T. N. Shellac	1·5272	1·5248	1·5225	1·5190	1·5140	1·5100	1·5060	1·5020
T. N. Shellac F. O.	1·5246	1·5230	1·5214	1·5182	1·5145	1·5102	1·5052	1·5010
T. N. Shellac 12%	1·5300	1·5276	1·5250	1·5220	1·5175	1·5126	1·5078	1·5032
Garnet lac	1·5292	1·5275	1·5255	1·5230	1·5190	1·5150	1·5110	1·5070

TABLE II

Samples	Refractive Index at							
	20° C	30° C	40° C	50° C	60° C	70° C	80° C	90° C
Pure lac resin (E.I.)	1·5248	1·5232	1·5218	1·5202	1·5180	1·5156	1·5116	1·5072
Soft lac resin (E.S.)	1·4976	1·4938	1·4900	1·4860	1·4820	1·4780	1·4740	1·4702
Lac Wax	1·4910	1·4880	1·4830	1·4710	1·4564	1·4500	1·4462	1·4430
Gum Elemi	1·5330	1·5285	1·5240	1·5200	1·5160	1·5130	1·5090	1·5055
Gum Dammar	1·5350	1·5340	1·5330	1·5320	1·5305	1·5290	1·5250	1·5210
Teglac No. 15	1·5540	1·5522	1·5500	1·5470	1·5430	1·5400	1·5362	1·5324

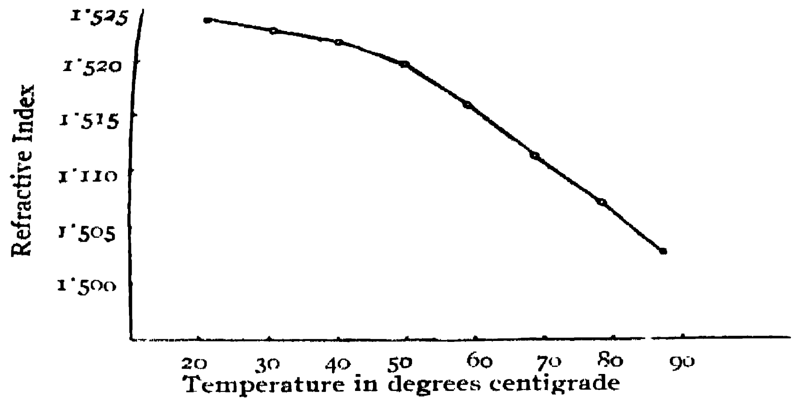
TABLE III

Lac sample	Author's data at 20°C	Data collected from other sources		
		Ref. Index	Temp.	Reference
Kusmi shellac	1·5224	1·515—1·518 1·520	23° C —	Thakur & Aldis Verman
Palas shellac	1·5235	1·517 1·516—1·519	18° C 23° C	Hanstock Thakur & Aldis
Dewaxed shellac	1·5228	1·521—1·522 1·519	23° C —	Thakur & Aldis Verman
Garnet lac	1·5295	1·520	18° C	Hanstock
T. N. Shellac	1·5272	1·513 1·54	18° C —	Hanstock Chamot & Mason
E. S. Resin	1·4976	1·503	23° C	Thakur & Aldis
E. I. Resin	1·5248	1·526 1·520	23° C —	Thakur & Aldis Verman
Shellac	1.5210—1·5336	1·524	—	Houwink
Orange superfine shellac	1·5244	1·516	—	Bradley

TABLE IV

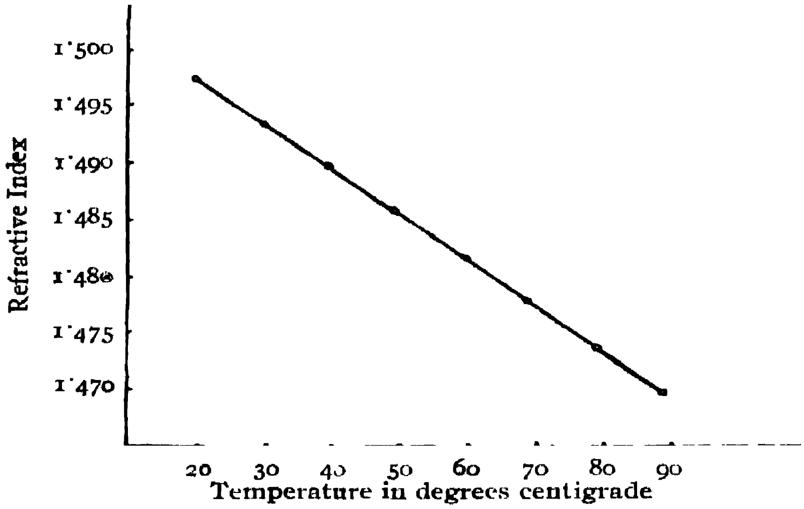
Temperature coefficients for lac and other resins

Samples	Temperature coefficient	Range of temperature in °C
Kusum shellac	−0·000175 −0·000300	Bet. 20 and 40 „ 70 and 90
Palas shellac	−0·000200 −0·000433	„ 20 and 40 „ 60 and 90
Khair shellac	−0·000175 −0·000433	„ 20 and 40 „ 60 and 90
Ber shellac	−0·000112 −0·000420	„ 20 and 40 „ 60 and 90
Pakur shellac	−0·00015 −0·000400	„ 20 and 40 „ 50 and 90
Button lac (R. L.)	−0·000210 −0·000400	„ 50 and 40 „ 50 and 90
Dewaxed shellac Blonde	−0·000200 −0·000400	„ 20 and 30 „ 40 and 90
Shellac Standard I	−0·000190 −0·000400	„ 20 and 40 „ 60 and 90
Shellac (Superfine unarsenicated)	−0·000210 −0·000400	„ 20 and 40 „ 60 and 90
Shellac (Superfine arsenicated)	−0·000200 −c·000410	„ 20 and 40 „ 50 and 90
Shellac (Fine Grade)	−0·000200 −0·000400	„ 20 and 40 „ 50 and 90
T. N. Shellac	−0·000240 −0·000400	„ 20 and 40 „ 60 and 90
T. N. Shellac F. O.	−0·000162 −0·000450	„ 20 and 40 „ 60 and 90
T. N. Shellac 12%	−0·000250 −0·000467	„ 20 and 40 „ 60 and 90
Garnet lac	−0·000200 −0·000400	„ 20 and 40 „ 50 and 90
Pure lac resin	−0·000167 −0·000425	„ 20 and 50 „ 70 and 90
Soft lac resin	−0·000400	„ 20 and 90
Lac wax	−0·000150 −0·000200	„ 20 and 30 „ 60 and 90
Gum Elemi	−0·000450 −0·000320	„ 20 and 40 „ 60 and 90
Gum Dammar	−0·000100 −0·000400	„ 20 and 50 „ 70 and 90
Teglac No. 15	−0·000200 −0·000375	„ 20 and 40 „ 50 and 90



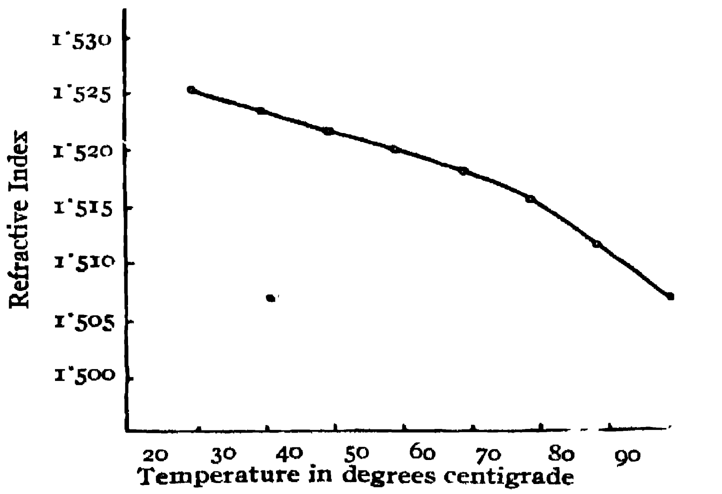
Refractive Index— Temp. Curve for Ber (*Zizyphus Jujuta*) lac

FIGURE 1



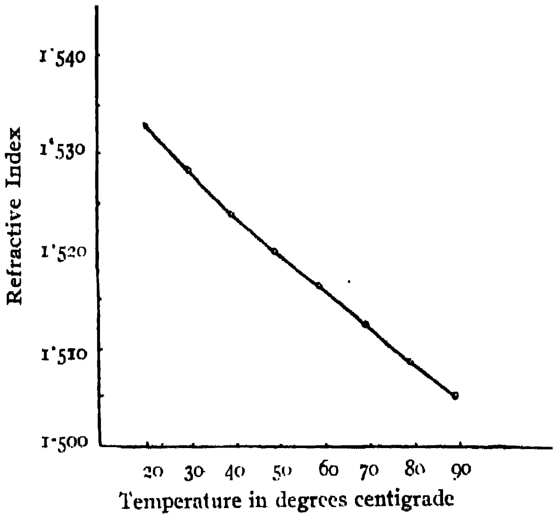
Refractive Index—Temp. Curve for soft lac resin

FIGURE 2



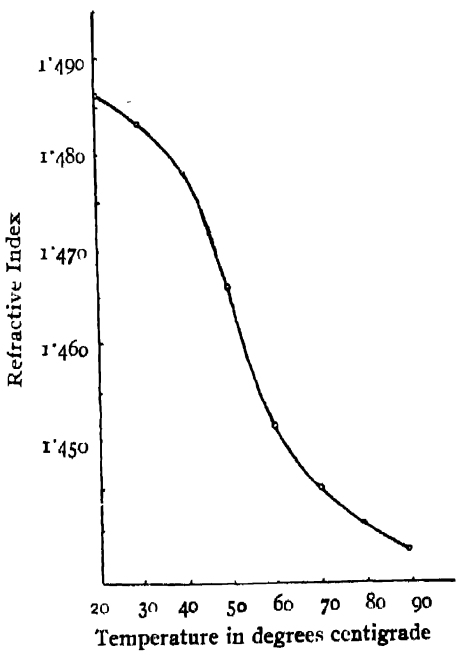
Refractive Index—Temperature Curve for pure lac resin

FIGURE 3



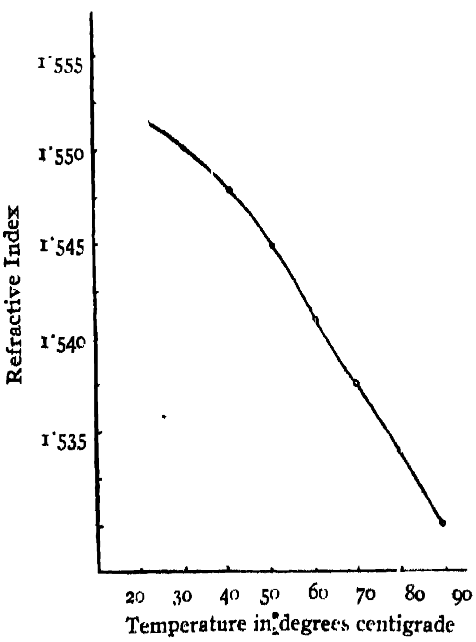
Refractive Index—Temp. Curve for Gum Elemi.

FIGURE 4



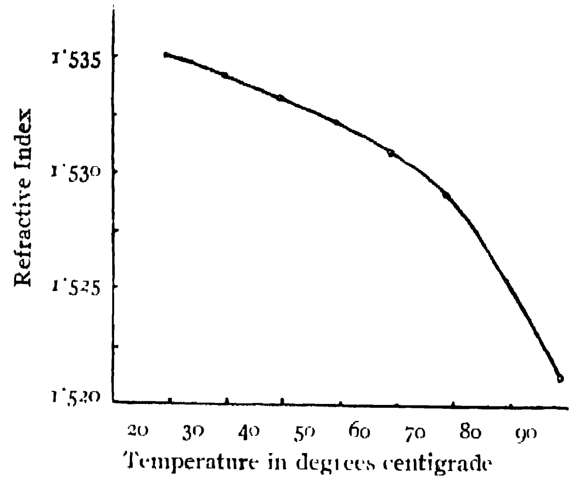
Refractive Index—Temp. Curve for Shellac Wax

FIGURE 5



Refractive Index—Temp. Curve for Teglac No. 15

FIGURE 6



Refractive Index—Temp. Curve for Dammar Gum

FIGURE 7

DISCUSSION

A typical curve representing the temperature variation of refractive index of lac has been shown in Figure 1. The graph is for Ber lac but most of the different varieties of lac examined fall under this group. From this curve it will be seen that there is a bend nearabout the region of 40°C—50°C. This simply shows that the temperature coefficients of refractive indices of lac change nearabout this range, indicating thereby the softening of the substance. The variation of refractive index is almost linear up to the lower limit of this softening range and it follows the same law after the higher limit, too. The soft resin constituent of lac, however, does not show any such bend on its curve, but follows a linear relationship throughout the range of investigation (Figure 2). It is easy to see, therefore, that the soft resin is a pure liquid even at 20°C and remains so throughout the range of temperature of investigation.

The pure resin, on the other hand, softens after 60°C and becomes liquid only after 80°C. (Figure 3).

Thus it is seen that the shellac or the pure resin begins actually to soften at a lower temperature than that found by the standard methods[9] generally used for the determination of the softening point. This fact has also been evidenced by the variation of the specific heat of lac with temperature.[10]

It may be easily seen that the Hanstock lens method has given somewhat lower values for the refactive index than the other methods. This was observed by Verman and he attributed the cause, at least partially, to the use of white light instead of sodium light. This explanation seems reasonable, for, although the lens apparatus was calibrated using a liquid whose refractive index was assumed for sodium light, it could not compensate completely for this discrepancy owing to the difference in the dispersion co-efficient of a resin and a liquid. That may, therefore, be the reason why the values obtained for refractive index of shellac by Hanstock or Verman or Thakur and Aldis are slightly lower than that found by Houwink or Chamot and Mason[11] or the present author. Houwink's value for the refractive index of shellac obtained by the immersion method coincides admirably with the average value for shellac obtained by the present method using Abbe Refractometer. For a few other resins, too, (both natural and synthetic) whose refractive indices have been measured with the Abbe Refractometer, the values are slightly different from those obtained by the lens method. Dammar Gum and Teg lac No. 15 give slightly higher values whilst the value for Gum Elemi is a bit lower than that obtained by Hanstock. It appears, however, that those substances which are liquid at ordinary temperatures have given slightly lower values by the Abbe Refractometer than those obtained by the lens method (cf. soft resin and Gum Elemi), whilst reverse is the case for other resins. This again suggests that the discrepancy is chiefly due to using white light instead of sodium light.

The temperature coefficient for almost all varieties of shellac is between 1.5×10^{-4} and 2.0×10^{-4} before the softening range and between 3.0×10^{-4} and 4.2×10^{-4} after this range. The average temperature co-efficient for the soft resin and the Gum Elemi is 4.0×10^{-4} throughout the temperature range of investigation. These co-efficients have been shown in a separate table. (Table IV).

The determination of the refractive indices of resins using Abbe Refractometer is, therefore, recommended either before the softening range starts, *i.e.*, below 40°C or after it ends, *viz.*, at about 70°C. The exact softening range will of course depend upon the nature of the particular resin of which the refractive index is to be determined. For all varieties of shellac, however, the higher temperature, *viz.*, about 70°C is very much desirable for the reason that it gives a very sharp, well defined line of demarcation between the two halves of the field of view and takes very much less time in the determination of this constant for routine work. For, this temperature, being near the melting point of practically all varieties of shellac, will serve both for the purpose of film formation on the lower prism of the refractometer as stated before, as well as for the determination of refractive indices. Determination at lower temperatures will mean raising first of all the temperature of the prism to a higher temperature for producing films and then lowering it for actual measurement. For cleaning the prisms again the temperature will have to be raised. As this naturally takes some time for each sample, it is an advantage to measure refractive indices of shellac or other low melting resins at a higher temperature. For hard lac resin or some other polymerised or adulterated lac samples it may be sometimes necessary to carry on the determination at 80°C or so. From the table it will appear that almost all genuine samples of lac will give a value within the range 1.505 to 1.511 at 70°C and within 1.501 to 1.507 at 80°C. Admixture of rosin always gives a higher value, as will be seen from the values obtained for a sample of shellac marked T.N. 12, containing 12% rosin. Thakur and Aldis similarly obtained a higher value for a sample containing 30% rosin.

Houwink has observed that resins generally give a higher value of refractive index on polymerisation. For a sample of shellac the value increased from 1.524 to 1.535 on heating for 100 hours at 110°C. Verman, on the other hand, found a definite decrease in the refractive index for trichlorethylene-extracted hard lac resin on heating overnight at 120°C. The present author, therefore, tried to determine the refractive index of heat hardened pure lac resin for confirmation of the point. It was found that ether-extracted pure lac resin on being heated at about 100°C for only 6 hours gave a slightly higher value of refractive index, *viz*., an increased value from 1.5248 to 1.5270.

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